

FORM PTO-1390
(REV 12-29-99)

U S DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

VER-148XX

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 36 U.S.C. 371

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

09/830907

INTERNATIONAL APPLICATION NO.

PCT/NL99/00676

INTERNATIONAL FILING DATE

04 November 1999

PRIORITY DATE CLAIMED

04 November 1998

TITLE OF INVENTION

STAR SHAPED ALUMINA EXTRUDATES AND CATALYST BASED THEREON

APPLICANT(S) FOR DO/EO/US

CORNELIS ROELAND BAYENSE; DURK YKEMA

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☐ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2)) in English
 - a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau). Published appln. WO 00/25918
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
 ☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information:
INFORMAL DRAWING (1 Sheet)

Express Mail Number

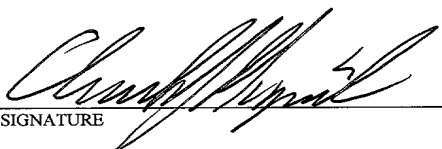
EL 634464636 US

U.S. APPLICATION NO. (If known, see 37 CFR 1.5) 09/830907		INTERNATIONAL APPLICATION NO PCT/NL99/00676		ATTORNEY'S DOCKET NUMBER VER-148XX	
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<p>17. <input checked="" type="checkbox"/> The following fees are submitted: BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)): Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO \$1,000.00</p> <p>International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO \$860.00</p> <p>International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International search fee (37 CFR 1.445(a)(2)) paid to USPTO \$710.00</p> <p>International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$690.00</p> <p>International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) \$100.00</p> <p style="text-align: right;">ENTER APPROPRIATE BASIC FEE AMOUNT =</p>	CALCULATIONS PTO USE ONLY																																							
<p>Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).</p> <table border="1" style="width:100%; border-collapse: collapse;"> <thead> <tr> <th style="width:20%;">CLAIMS</th> <th style="width:20%;">NUMBER FILED</th> <th style="width:20%;">NUMBER EXTRA</th> <th style="width:20%;">RATE</th> <th style="width:20%;"></th> <th style="width:20%;"></th> </tr> </thead> <tbody> <tr> <td>Total claims</td> <td>19 - 20 =</td> <td>0</td> <td>X \$18.00</td> <td>\$0</td> <td></td> </tr> <tr> <td>Independent claims</td> <td>1 - 3 =</td> <td>0</td> <td>X \$80.00</td> <td>\$0</td> <td></td> </tr> <tr> <td colspan="4">MULTIPLE DEPENDENT CLAIM(S) (if applicable)</td> <td>+\$270.00</td> <td>\$-</td> </tr> <tr> <td colspan="4" style="text-align: right;">TOTAL OF ABOVE CALCULATIONS =</td> <td>\$860.00</td> <td></td> </tr> </tbody> </table> <p><input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.</p> <p style="text-align: right;">SUBTOTAL =</p> <p>Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).</p> <p style="text-align: right;">TOTAL NATIONAL FEE =</p> <p>Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property</p> <p style="text-align: right;">TOTAL FEES ENCLOSED =</p>	CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE			Total claims	19 - 20 =	0	X \$18.00	\$0		Independent claims	1 - 3 =	0	X \$80.00	\$0		MULTIPLE DEPENDENT CLAIM(S) (if applicable)				+ \$270.00	\$-	TOTAL OF ABOVE CALCULATIONS =				\$860.00		<table border="1" style="width:100%; border-collapse: collapse;"> <tr> <td style="width:50%;"></td> <td style="width:50%; text-align: center;">Amount to be Refunded</td> <td style="width:10%; text-align: center;">\$</td> </tr> <tr> <td></td> <td style="text-align: center;">:</td> <td></td> </tr> <tr> <td></td> <td style="text-align: center;">Charged:</td> <td style="text-align: center;">\$</td> </tr> </table>		Amount to be Refunded	\$:			Charged:	\$
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Independent claims	1 - 3 =	0	X \$80.00	\$0																																				
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a. <input checked="" type="checkbox"/>	A check in the amount of <u>\$860.00</u> to cover the above fees is enclosed. A check in the amount of \$_____ is enclosed for the assignment recordation fee.
b. <input type="checkbox"/>	Please charge my Deposit Account No. _____ in the amount of \$_____ to cover the above fees. A duplicate copy of this sheet is enclosed.
c. <input checked="" type="checkbox"/>	The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>23-0804</u> . A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

<p>Customer Number 207</p> <p>SEND ALL CORRESPONDENCE TO:</p> <p>Weingarten, Schurgin, Gagnebin & Hayes LLP Ten Post Office Square Boston, Massachusetts 02109</p> <p>Date: <u>5-2-1</u></p>	 SIGNATURE <u>Charles L. Gagnebin III</u> NAME <u>25,467</u> REGISTRATION NUMBER
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application : CORNELIS ROELAND BAYENSE, ET AL.
Application No. :
Filed : Herewith
For : STAR SHAPED ALUMINA EXTRUDATES AND
CATALYST BASED THEREON
Examiner :
Attorney's Docket : VER-148XX

Group Art Unit:

* * * * *
I hereby certify that this correspondence is being deposited
with the United States Postal Service as first class mail in an
envelope addressed to: Assistant Commissioner for Patents,
Washington, D.C. 20231 on _____.

By: _____
Charles L. Gagnebin III
Registration No. 25,467
Attorney for Applicant(s)

* * * * *

PRELIMINARY AMENDMENT

BOX PCT
Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

Kindly enter the following Preliminary Amendment in the
above-identified application:

Express Mail Number

EL 634464636 US

In the Claims:

Please amend the Claims to read as follows (a copy of the amended claims showing the additions and deletions appears at the end for the Examiner's convenience):

3. Extrudates according to claim 1, having a length to diameter ratio of between 1 and 3.

4. Extrudates according to claim 1, wherein the total pore volume as determined by mercury porosimetry is between 0.5 and 0.75 ml/g.

5. Extrudates according to claim 1, wherein the BET surface area is at least 75 m²/g.

6. Extrudates according to claim 1, wherein the attrition in accordance with ASTM D4058-87 is less than 5 wt.%, preferably less than 3 wt.%.

7. Catalyst, comprising at least one catalytically active material supported on an extrudate according to claim 1.

9. Use of an extrudate according to claim 1 in a chemical reaction.

Please add the following new claims 10 - 19:

10. Extrudates according to claim 2, having a length to diameter ratio of between 1 and 3.

11. Extrudates according to claim 10, wherein:

the total pore volume as determined by mercury porosimetry is between 0.5 and 0.75 ml/g;

the BET surface area is at least 75 m²/g; and

the attrition in accordance with ASTM D4058-87 is less than 5 wt.%, preferably less than 3 wt.%.

12. Catalyst, comprising at least one catalytically active material supported on an extrudate according to claim 2.

13. Catalyst, comprising at least one catalytically active material supported on an extrudate according to claim 3.

14. Catalyst, comprising at least one catalytically active material supported on an extrudate according to claim 4.

15. Catalyst, comprising at least one catalytically active material supported on an extrudate according to claim 5.

16. Catalyst, comprising at least one catalytically active material supported on an extrudate according to claim 6.

17. Use of an extrudate according to claim 11 in a chemical reaction.

18. Use of a catalyst according to claim 7 in a chemical reaction.

19. Use of a catalyst according to claim 8 in a chemical reaction.

REMARKS

This Preliminary Amendment puts the claims into proper form for examination. Note that claims 3-7 and 9 have been amended; new claims 10-19 have been added; and claims 1, 2, and 8 remain


Attorney Docket No. VER-148XX
Filed: Herewith
Group Art Unit:

unchanged. Kindly calculate the filing fee based on the amended claims.

The Examiner is encouraged to telephone the undersigned attorney to discuss any matter which would expedite allowance of the present application.

Respectfully submitted,

CORNELIS ROELAND BAYENSE,
ET AL.

By: 
Charles L. Gagnebin III
Registration No. 25,467
Attorney for Applicants

WEINGARTEN, SCHURGIN, GAGNEBIN
& HAYES LLP
Ten Post Office Square
Boston, MA 02109
Telephone: (617) 542-2290
Telecopier: (617) 451-0313

Date: 5.21

CLG/mc/248831-1
Enclosures

Red-lined Claims for the Examiner's convenience

3. Extrudates according to claim 1~~or~~2, having a length to diameter ratio of between 1 and 3.

4. Extrudates according to claims 1-3, wherein the total pore volume as determined by mercury porosimetry is between 0.5 and 0.75 ml/g.

5. Extrudates according to claims 1-4, wherein the BET surface area is at least 75 m²/g.

6. Extrudates according to claims 1-5, wherein the attrition in accordance with ASTM D4058-87 is less than 5 wt.%, preferably less than 3 wt.%.

7. Catalyst, comprising at least one catalytically active material supported on an extrudate according to claims 1-6.

9. Use of an extrudate according to claims 1-6~~or a catalyst according to claim 7 or 8~~ in a chemical reaction.

Title: STAR SHAPED ALUMINA EXTRUDATES AND CATALYST BASED THEREON

The invention is directed to transition alumina extrudates, suitable as catalyst, or as catalyst support, and the use of such extrudates in chemical reactions.

In catalysis alumina plays an important role, both as a catalyst support and as catalytically active material. As is mentioned in Kirk-Othmer, Third Ed, Vol. 2, pages 230-232, alumina can be used as catalyst in a Claus process, for dehydration of alcohols, such as the production of olefins from alcohol, and the reverse reaction, but also for the isomerisation of olefins. As interacting catalyst support alumina may play a role in hydrotreating catalysts, e.g. in cobalt or nickel-molybdenum oxides on alumina.

As a support alumina is frequently used for precious metal catalyst, such as in exhaust catalysts or for (de)hydrogenation reactions. As support for a nickel catalyst it may be used in (de)hydrogenation reactions such as for fat and oils hydrogenation, for hydrogenation of fatty nitriles or of nitro aromatic compounds or for oligomerisation of olefins.

The structure of the support, i.e. the BET surface area, the pore size and the pore volume distribution, forms an important aspect of the alumina or alumina based catalyst. In view of activity and selectivity it would be highly desirable to have an alumina product that is on the one hand highly porous, i.e. having a large volume in large pores, and that has a good mechanical strength and stability. Unfortunately these are requirements that are difficult to reconcile with each other.

In fixed bed processes shaped bodies of alumina are frequently used. An important aspect therein is the shape dependency of the pressure drop. Tablets and extrudates are the materials commonly used in fixed bed applications. In

New Page 2

order to minimise pressure drop, the use of star shaped extrudates would be most suitable. However, star shaped bodies, extrudates, tend to be prone to attrition due to the presence of the 'points' of the star.

EP-A-0 008 424 discloses star shaped desulfurization catalysts.

- 5 Highly porous star shaped catalysts, having a good mechanical strength and stability are not disclosed.

It is an object of the invention to reconcile these various requirements in the form of a transition alumina extrudate, having a carefully balanced set of properties. Further objects and advantages will become clear from the following description of the invention and the preferred embodiments thereof.

10

The invention is based thereon that the inventors have now been able to provide a star shaped alumina extrudate, having on the one hand an optimal structure, as indicated above and on the other hand a good strength.

- 15 The invention is accordingly directed to star shaped alumina extrudates with a pore volume in pores of diameter of over 1000 nm, as determined by mercury porosimetry, of at least 0.05 ml/g, a side crushing strength of at least 50 N and a bulk crushing strength of at least 1 MPa.

- 20 Surprisingly, this set of properties can be made available in one material, thereby providing a material with which chemical reactions can be made much more efficient, resulting in higher activity and/or selectivity. Also the material of the invention, when used in fixed bed reactors, provides a decreased pressure drop compared to regular extrudates having a cylindrical shape.

- 25 The BET surface area, as determined by single point adsorption using the BET equation (as e.g. described by G. Sandstede et.al, Chem. Ing. Tech. 32 (1960), 413), should be at least 10 m²/g of alumina. This coincides with the requirement of using a transition alumina, i.e. not an α alumina. Suitable alumina's are the various transition alumina's including γ -alumina, δ -alumina, ϵ -alumina, κ -alumina, ζ -alumina, θ -alumina and τ -alumina. These alumina's have a large BET-surface area, generally in the range of 25 up to more than 100 m²/g.
- 30

The pore volume is a further important requirement, whereby it is on the one hand important that the total pore volume, as determined by mercury intrusion is sufficiently high and on the other hand that the pore volume in pores of over 1000 nm forms a substantial portion of the total pore volume. In absolute terms the total pore volume should be at least 0.50 ml/g, whereas the ratio of the pore volume in pores of over 1000 nm to total pore volume should preferably be more than 0.04. An alumina having those properties has good properties in terms of reactant accessibility, which makes it very suitable for all kinds of catalytic reactions requiring good diffusion of reactants and products through the alumina, thereby eliminating diffusion limitation problems as much as possible.

The pore volume and pore size distribution are determined by mercury porosimetry measurements, as described by J. Rouquerol et al in Pure & Applied Chem., 66(8),1994, pages 1752-1753, using the Washburn equation.

As indicated above, the use of star shaped extrudates is important in terms of pressure drop in relation to accessibility of the internal surface of the alumina. This also plays a role in eliminating diffusion problems. Star shaped extrudates can be defined as objects having some kind of central part or core, with three or more triangularly shaped extensions on the circumference thereof. Most preferred are star shaped extrusions having five extensions, as this provides the optimal balance between strength, porosity, pressure drop and accessibility. Another advantageous property of the star shaped extrudates is the fact that the ratio of external surface area to volume is more advantageous than in the case of conventional cylindrical extrudates or tablets.

The ratio of the length of the extrudates to the diameter is preferably between 1 and 3, whereby as diameter the distance is meant between two parallel planes on either side of the extrudate.

Important aspects of the material of the invention are also the strength characteristics. As indicated above a side crushing strength of at least 50 N and a bulk crushing strength of at least 1 MPa are essential herein. These parameters form the basis for the suitability of the extrudates for use in large scale reactors, like in the petroleum industry. When the extrudates meet these requirements, they can be used in huge fixed bed reactors, that require very strong material. The side crushing strength and the bulk crushing strength is defined as follows:

The side crushing strength (SCS) of extrudates is defined as the pressure (in Newtons) at which extrudates of 4.5-5.00 mm length are crushed, when treated under pressure between two flat plates on a AIKOH, 9500 series tester.

The bulk crushing strength (BCS) of a catalyst is defined as the pressure (in Megapascals) at which 0.5% fines (i.e. particles less than 0.425 mm) are formed when treated under a piston in a tube. For that purpose, 17 ml of catalyst particles, presieved on a 0.425 mm sieve, are loaded in a cylindrical sample tube (diameter 27.3 mm), and 8 ml steel beads is loaded on top. The catalyst is subsequently treated at different (increasing) pressures for three minutes, after which the fines are recovered and their percentages is determined. This procedure is repeated until a level of 0.5 wt.% fines is reached.

Another aspect of the strength of the material is the attrition, i.e. the amount of material that may break off of the extrudates upon use. This attrition, determined in accordance with ASTM D4058-87, should preferably be less than 5 wt.%, more in particular less than 3 wt.%.

The alumina extrudates having the above properties can be prepared by mixing transition alumina powder with a suitable binder in the presence of a liquid, usually water or an aqueous solution of a mineral acid such as hydrochloric, sulfonic or nitric acid, to form a paste, followed by extruding of the paste in the required star form, using a

suitable die and cutting the extruded strands of material to the required length. Optionally after drying, the extrudates are calcined.

It is possible to use various types of binder materials, such as those based on silica or alumina. Examples are colloidal silica, waterglass, or clays. It is preferred to use an alumina based binder or a binder that is removed during calcination, while providing and maintaining the required strength. An example of a suitable binder system is an alumina binder that gels under acidic treatment, for example by using organic or inorganic acids. The amount of binder material used in the preparation of the paste that is to be extruded will vary depending on the type of material and the required strength. Generally it will not be in excess of 30 wt.% based on the dry weight of binder and alumina together.

The invention will now be elucidated on the basis of an example.

EXAMPLE

1.5kg of aluminium trihydrate, containing 65 wt.% of Al_2O_3 , with an average particle size of 30-50 μm is mixed with 0.4 kg of alumina binder. The powders are mixed extensively while slowly adding diluted, aqueous HNO_3 in an amount of 2 wt.%, calculated on the weight of the total amount of alumina.

Thereby the alumina binder is peptised. Mixing is continued until a relatively dry product is obtained. the intermediate product is extruded using a one-screw extruder, equipped with a die having starshaped holes and a cutting device.

The extrudates obtained are dried at 105°C for 16 hours and subsequently calcined at 850°C for one hour. Attached are two figures with photographs of an extrudate shown from two different angles.

N₂-BET surface area 106 m/g²

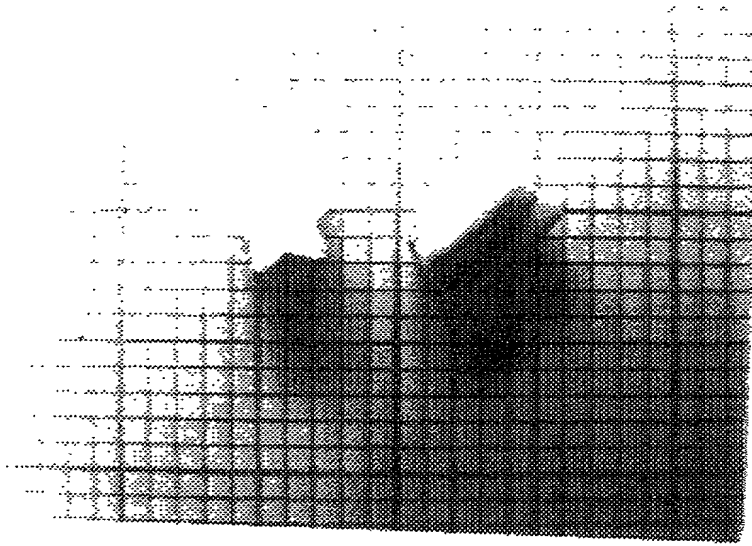
5 Pore volume in pores over 1000 nm 0.07 ml/g

Bulk crushing strength 1.08 MPa

Claims

1. Star shaped alumina extrudates with a pore volume in pores of diameter of over 1000 nm, as determined by mercury porosimetry, of at least 0.05 ml/g, a side crushing strength of at least 50 N and a bulk crushing strength of at least
- 5 1 MPa.
2. Extrudates according to claim 1, having a length of between 2 and 8mm.
3. Extrudates according to claim 1 or 2, having a length to diameter ratio of between 1 and 3.
- 10 4. Extrudates according to claims 1-3, wherein the total pore volume a determined by mercury porosimetry is between 0.5 and 0.75 ml/g.
5. Extrudates according to claims 1-4, wherein the BET surface area is at least 75 m²/g.
- 15 6. Extrudates according to claims 1-5, wherein the attrition in accordance with ASTM D4058-87 is less than 5 wt.%, preferably less than 3 wt.%.
7. Catalyst, comprising at least one catalytically active material supported on an extrudate according to claims
- 20 1-6.
8. Catalyst according to claim 7, wherein the catalytically active material is selected from the group of metals, metal oxides, metal sulfides and combinations thereof.
- 25 9. Use of an extrudate according to claims 1-6 or a catalyst according to claim 7 or 8 in a chemical reaction.

09/830907-084904



DECLARATION AND POWER OF ATTORNEY

As a below-named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name:

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled: STAR SHAPED ALUMINA EXTRUDATES AND CATALYST BASED THEREON

the specification of which (check one):

[] is attached hereto. [X] was filed 05/02/2000 as Application No. 09/830,907
amended on _____ (if applicable).

[X] was filed as PCT International Application No. PCT/NL99/00676 on 4 November 1999,
and was amended under PCT Article 19 on (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations §1.56(a).

I hereby claim foreign priority benefits under Title 35, USC §119(a)-(d) of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

<u>Prior Foreign Application(s)</u>		<u>Date Filed</u>	<u>Priority Claimed</u>	
98 203719.4	Europe	4 November 1998	[X]	[]
(Number)	(Country)	(Day/Month/Year)	Yes	No
			[]	[]
(Number)	(Country)	(Day/Month/Year)	Yes	No

I hereby claim the benefit under Title 35, USC §119(e) of any United States provisional application(s) listed below:

(Application Number)

(Filing Date)

(Application Number)

(Filing Date)

(Application Number)

(Filing Date)

Express Mail Number

EL 63446497605

Attorney

Docket No.: VER-148XX

I hereby claim the benefit under Title 35 USC §120 of any United States application(s) listed below and insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35 USC §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application No.)	(Filing Date)	(Patented/pending/abandoned)
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(Application No.)	(Filing Date)	(Patented/pending/abandoned)
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POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) to prosecute this application and transact all business connected therewith in the Patent and Trademark Office, and to file with the USRO any International Application based thereon.

Stanley M. Schurgin, Reg. No. 20,979
 Charles L. Gagnebin III, Reg. No. 25,467
 Paul J. Hayes, Reg. No. 28,307
 Victor B. Lebovici, Reg. No. 30,864

Eugene A. Feher, Reg. No. 33,171
 Beverly E. Hjorth, Reg. No. 32,033
 Holliday C. Heine, Reg. No. 34,346
 Gordon R. Moriarty, Reg. No. 38,973
 James F. Thompson, Reg. No. 36,699

Address all correspondence to:

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Ten Post Office Square

Boston, Massachusetts 02109

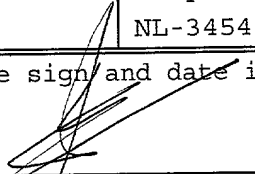
Telephone: (617) 542-2290

Telecopier: (617) 451-0313

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full Name of Sole/First Inventor: <u>Cornelis Roeland Bayense</u>		
City of Residence <u>Gameren</u>	State or Country <u>Netherlands</u> <i>NLX</i>	Country of Citizenship <u>Netherlands</u>
Post Office Address <u>Delkant 6</u>	City <u>NL-5311 CJ Gameren</u>	State or Country Zip Code <u>Netherlands</u>
Signature: (Please sign and date in permanent ink.) X <i>CR Bayense</i>		Date signed: X <i>14 - June - '01</i>

Attorney
Docket No.: VER-148XX

Full Name of Second Joint Inventor: <u>Durk Ykema</u>		
City of Residence <u>De Meern</u>	State or Country Netherlands <u>NL-X</u>	Country of Citizenship Netherlands
Post Office Address <u>Hemelvuur 13</u>	City NL-3454 SP De Meern	State or Country Zip Code Netherlands
Signature: (Please sign and date in permanent ink.)  X		Date signed: X <u>14-JUNE-2001</u>